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on December 19, 2003

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12/19/03
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Signature

PATENT
#02-0483-UNI
Case #C4247(C)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: del Nunzio et al.
Serial No.: 10/664,370
Filed: September 17, 2003
For: DETERGENT COMPOSITIONS

Edgewater, New Jersey 07020
December 19, 2003

SUBMISSION OF PRIORITY DOCUMENT

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Pursuant to rule 55(b) of the Rules of Practice in Patent Cases, Applicant(s) is/are submitting herewith a certified copy of the United Kingdom Application No. 0221735.4 filed September 19, 2002, upon which the claim for priority under 35 U.S.C. § 119 was made in the United States.

It is respectfully requested that the priority document be made part of the file history.

Respectfully submitted,

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INVESTOR IN PEOPLE

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I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears an amendment, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

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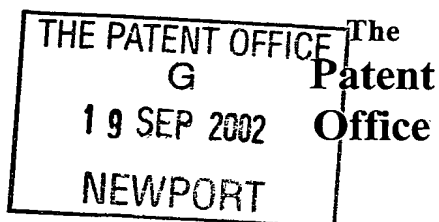
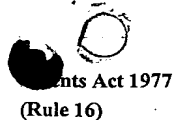
Stephen Hordley

Dated

15 August 2003

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Patents Form 1/77


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 P01/7700 0-00-0221735.4

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Request for grant of a patent

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- | | | | | | |
|-------------------------------|---|--|-------------------------------|--|---|
| 1. | Your reference | C4247 (C)/rkk | | | |
| 2. | 0221735.4 | 19 SEP 2002 | | | |
| 3. | Full name, address and postcode of the or of each applicant (<i>underline all surnames</i>) | UNILEVER PLC
UNILEVER HOUSE, BLACKFRIARS
LONDON, EC4P 4BQ | | | |
| | Patents ADP number (<i>if you know it</i>) | 50426956002 ✓ | | | |
| | If the applicant is a corporate body, give the country/state of its incorporation | UNITED KINGDOM | | | |
| 4. | Title of the invention | DETERGENT COMPOSITIONS | | | |
| 5. | Name of your agent (<i>if you have one</i>) | FRANSELLA MARY
Ellioft. Peter William | | | |
| | "Address for Service" in the United Kingdom to which all correspondence should be sent (<i>including the postcode</i>) | PATENT DEPARTMENT, UNILEVER PLC
COLWORTH HOUSE, SHARNBROOK
BEDFORD, MK44 1LQ | | | |
| | Patents ADP number (<i>if you know it</i>) | 689344000 6572927001 | | | |
| 6. | If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (<i>if you know it</i>) the or each application number | <table border="0"> <tr> <td>Country</td> <td>Priority application number
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| 7. | If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application | <table border="0"> <tr> <td>Number of earlier application</td> <td>Date of filing
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| 8. | Is a statement of inventorship and of right to grant of a patent required in support of this request? (<i>Answer 'Yes' if:</i>
a) <i>any applicant named in part 3 is not an inventor, or</i>
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Translations of priority documents

Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination and search (*Patents Form 9/77*)

Request for substantive examination (*Patents Form 10/77*)

Any other documents
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11. I/We request the grant of a patent on the basis of this application.

Signature(s)

Date: 18/09/02

Sandra Jane EDWARDS, Authorised Signatory

12. Name and daytime telephone number of person to contact in the United Kingdom

Ravinder K Kundra, Tel 01234 22 2439

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DETERGENT COMPOSITIONSTECHNICAL FIELD

- 5 The present invention relates to particulate detergent compositions containing effervescent granules.

BACKGROUND AND PRIOR ART

- 10 Particulate laundry detergent compositions must satisfy a wide range of practical constraints. As well as providing excellent cleaning and care, they must also satisfy numerous physical constraints which go beyond this basic requirement. For example it is known that the effervescence properties of
- 15 certain added ingredients can provide improved dispensing and dispersing qualities to the detergent composition. This can be achieved by adding two ingredients which react together in the presence of water to produce a gas, for example by using sodium carbonate and citric acid. It is
- 20 also known that improved effervescence may result if these two reacting components are placed together in a single granule. Such a single granule is often referred to in the art as an effervescent granule.
- 25 US 4 252 664 (Colgate-Palmolive) discloses the use of an effervescent granule comprising a gas-producing agent (preferably a carbonate) and an acid (preferably a solid acid). The effervescent granule is present in the full formulation at a level of from 5 to 10 wt%.

A series of patent applications have published which disclose effervescent granules which also contain detergent ingredients such as surfactant and builder:

- 5 WO 98 42811 (Procter & Gamble) discloses a detergent granule of size 1.0 to 4.5 mm which comprises an acid source and an alkali source.

WO 98 46714 (Procter & Gamble) discloses a substantially
10 anhydrous effervescent particle of size 0.075 to 20 mm.

WO 98 46715 (Procter & Gamble) discloses a substantially anhydrous foaming component comprising an anhydrous surface active component (e.g. surfactant) and an effervescence
15 component (e.g. acid or alkali source).

WO 98 46716 (Procter & Gamble) discloses a dry effervescent granule comprising an acid, a carbonate source and a binder, wherein the acid and carbonate source are in close physical
20 proximity.

More recently, it has been discovered that effervescent granules may be improved by controlling the particle sizes of its constituents:

25

WO 00 34422 (Procter & Gamble) discloses an effervescence component which comprises an acid source and a carbonate source wherein at least 75% of the acid source has a particle size of from 0.1 to 150 microns. Preferably the
30 carbonate source also has a defined particle size and the granule is preferably coated in a nonionic surfactant.

- 3 -

WO 01 30949 (Procter & Gamble) discloses a reactive particle comprising two particulate reactants in which the particle number ratio of the first reactant to the second is at least 50:1. Preferably the median particle size of the second to
5 the first reactant is at least 2:1.

However, a difficulty in using such effervescent granules is their tendency to prematurely react with moisture during storage which reduces their degree of effervescence when
10 added to a wash liquor.

WO 01 30953 (Procter & Gamble) discloses the combination of an effervescent granule and a low relative humidity base powder. The base powder is stated to act as a moisture sink
15 which prevents the effervescent granule from deactivating due to moisture.

Although a detergent composition with a low relative humidity may be used, this may place severe formulation
20 constraints upon it.

The present inventors have surprisingly found that an effervescent granule which comprises an acid source, a carbonate source and a desiccant provides an effervescent
25 granule which may be included in a wider range of detergent compositions than was previously thought possible and still maintain good dispensing properties.

STATEMENT OF INVENTION

The present invention provides a particulate laundry detergent composition comprising an effervescent granule,
5 the effervescent granule comprising an acid source, a carbonate source and a desiccant, wherein the detergent composition has an equilibrium relative humidity at 25°C and 1 atmosphere of at least 10%.

10 DETAILED DESCRIPTION OF THE INVENTIONThe Effervescent Granule

The effervescent granule of the present invention comprises
15 an alkali source, a carbonate source and a desiccant. The acid source and carbonate source are preferably in particulate form and therefore each granule would contain discrete particles of acid and carbonate source.

20 The granule preferably comprises from 20 to 80 wt%, preferably from 30 to 60 wt%, more preferably from 40 to 50 wt% of the carbonate source. The granule preferably comprises from 10 to 60, preferably from 20 to 50, more preferably from 30 to 40 wt% of the acid source.

25

It is preferred that the majority of the granule is comprised of the carbonate and the acid source and that more preferably the carbonate source and the acid source make up from 50 to 100 wt%, preferably from 60 to 99 wt%, more
30 preferably from 70 to 95 wt%, most preferably from 80 to 90 wt% of the granule.

The granules preferably have a particle size of from 0.2 to 10.0 mm, preferably from 0.5 to 5.0 mm, more preferably from 0.8 to 4.0 mm. This helps to provide a highly effective localised fizzing action.

5

The granule may also comprise a non-aqueous binder to help hold it together and it is preferred that such a binder is a polyethylene glycol, a polypropylene glycol or an alcohol.

- 10 The granules may also comprise a colourant in order to contrast their appearance from that of the detergent compositions to which they are added.

The Desiccant

15

The desiccant may be any suitable moisture absorbing desiccant which is soluble or dispersible in water. Suitable materials are an activated desiccant clay, activated alumina, a dried zeolite or silica gel.

- 20 Preferably silica gel is used.

The level of desiccant is suitably low and therefore is preferably present at a level of from 0.1 to 10 wt%, preferably from 0.2 to 5 wt%, more preferably from 0.3 to 2 wt%, of the granule.

25

The Acid Source

The acid source reacts with the carbonate source in the presence of water to produce carbon dioxide gas.

30

- 6 -

Preferably the acid source is a particulate material and has a narrow particle size distribution such that at least 50 wt%, preferably at least 60 wt%, more preferably at least 70 wt%, most preferably at least 80 wt% of particles have a size of from 150 to 350 microns.

Preferably the acid source is an organic acid. Preferably the acids are mono-, bi- or tri-protonic acids. Such preferred acids include mono- or polycarboxylic acids preferably citric acid, adipic acid, glutaric acid, citramalic acid, tartaric acid, maleic acid, malic acid, succinic acid, malonic acid. Such acids are preferably used in their acidic forms, and it may be preferred that their anhydrous forms are used, or mixtures thereof. Citric acid is the most highly preferred.

The Carbonate Source

The carbonate source reacts with the acid source in the presence of water to produce carbon dioxide gas.

The carbonate source is preferably selected from carbonate, bicarbonate, sesquicarbonate and mixtures thereof. A highly preferred carbonate source is sodium bicarbonate.

25

In one preferred embodiment the granule comprises sodium bicarbonate with a narrow particle size distribution such that at least 50 wt%, preferably at least 60 wt%, more preferably at least 70 wt%, most preferably at least 80 wt% of particles have a size of from 20 to 60, preferably 38 to 45 microns.

Sodium bicarbonate and sodium carbonate may be advantageously combined in a weight ratio of greater than 3:2, preferably at least 2:1, more preferably at least 3:1.

5 The Optional Solid Surfactant Particles

In order to enhance the fizzing action and to provide a positive cue for the end user, the effervescent granules of the present invention may comprise solid surfactant
10 particles. It is preferred that in its isolated state the surfactant is sufficiently solid such that it is pourable.

As desired, the granule may comprises from 1 to 15 wt%, preferably from 2 to 10 wt%, more preferably from 3 to 8 wt%
15 of solid surfactant particles.

Preferably the surfactant has a melting point of at least 30°C, preferably at least 40°C, more preferably at least 50°C.

20

Surfactants suitable for the present invention meet the criteria of being solid and particulate. Highly preferred surfactants are the alkyl sulphate type surfactants, preferably a C₈-C₁₅ primary alkyl sulphate, more preferably a
25 C₁₀-C₁₄ primary alkyl sulphate. Also preferred are the C₈-C₁₅ olefin sulphonates.

Preferably the granules are substantially free of alkyl benzene sulphonate surfactants, because of their
30 hygroscopicity. In this context, 'substantially free' means

less than 5 wt% of the effervescent granule, preferably less than 3 wt%, more preferably less than 1 wt%, most preferably completely absent.

- 5 Preferably the surfactant particles have a d₅₀ particle size of greater than 100 microns, preferably from 150 to 800 microns.

10 In a particularly preferred embodiment, the ratio of the particle size of the effervescent granule to the d₅₀ particle size of the surfactant is greater than 5:1, preferably from 6:1 to 1000:1.

The Method of Manufacture

15

The effervescent granules according to the present invention may be made by any suitable process, such as for example by granulation, roll-compaction, extrusion or a combination thereof.

20

The Detergent Composition

25 The effervescent granules of the present invention are intended to be added to a conventional particulate laundry detergent composition. Preferably they are present at a level of from 0.1 to less than 10 wt%, more preferably from 0.5 to less than 4 wt%, most preferably from 0.5 to less than 2 wt%.

The compositions of the invention are characterised by a relative humidity value at 1 atm and 25°C at least 10%. The term "relative humidity value" as used herein means the relative humidity of air in equilibrium with the

5 composition: it is an indirect measurement of the water activity in a solid. It is the ratio of the current water concentration in the air (kg water/kg air) to the maximum at a given temperature and pressure, expressed as a percentage of the value for saturated air. For a solid an equilibrium
10 is established between the water in the solid and the atmosphere, and the measured relative humidity is a characteristic for that solid at a given temperature and pressure. All relative humidity values quoted in the present specification are normalised to 1 atmosphere
15 pressure and 25°C.

Since the effervescent granules of the present invention comprise a desiccant, they are particularly suitable for detergent compositions with a high equilibrium relative
20 humidity. Hence detergent compositions according to the present invention have an equilibrium relative humidity value at 25°C of preferably at least 15%, more preferably at least 20%, most preferably at least 25%, or even greater than 30%. In spite of the high relative humidities of the
25 detergent composition, the effervescent granule exhibits acceptable storage stability.

Detergent compositions of the invention contain detergent-active compounds and detergency builders, and may optionally
30 contain bleaching components and other active ingredients to enhance performance and properties.

Detergent Ingredients

Detergent-active compounds (surfactants) may be chosen from
5 soap and non-soap anionic, cationic, nonionic, amphoteric
and zwitterionic detergent-active compounds, and mixtures
thereof. Many suitable detergent-active compounds are
available and are fully described in the literature, for
example, in "Surface-Active Agents and Detergents", Volumes
10 I and II, by Schwartz, Perry and Berch. The preferred
detergent-active compounds that can be used are soaps and
synthetic non-soap anionic and nonionic compounds. The
total amount of surfactant present is suitably within the
range of from 5 to 40 wt%.

15 Anionic surfactants are well-known to those skilled in the
art. Examples include alkylbenzene sulphonates,
particularly linear alkylbenzene sulphonates having an alkyl
chain length of C₈-C₁₅; primary and secondary
20 alkylsulphates, particularly C₈-C₁₅ primary alkyl sulphates;
alkyl ether sulphates; olefin sulphonates; alkyl xylene
sulphonates; dialkyl sulphosuccinates; and fatty acid
ester sulphonates. Sodium salts are generally preferred.

25 Nonionic surfactants that may be used include the primary
and secondary alcohol ethoxylates, especially the C₈-C₂₀
aliphatic alcohols ethoxylated with an average of from 1 to
20 moles of ethylene oxide per mole of alcohol, and more
especially the C₁₀-C₁₅ primary and secondary aliphatic
30 alcohols ethoxylated with an average of from 1 to 10 moles

of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

- 5 Cationic surfactants that may be used include quaternary ammonium salts of the general formula $R_1R_2R_3R_4N^+ X^-$ wherein the R groups are long or short hydrocarbyl chains, typically alkyl, hydroxyalkyl or ethoxylated alkyl groups, and X is a solubilising cation (for example, compounds in which R_1 is a
- 10 C_8 - C_{22} alkyl group, preferably a C_8 - C_{10} or C_{12} - C_{14} alkyl group, R_2 is a methyl group, and R_3 and R_4 , which may be the same or different, are methyl or hydroxyethyl groups); and cationic esters (for example, choline esters).
- 15 Detergent compositions suitable for use in most automatic fabric washing machines generally contain anionic non-soap surfactant, or nonionic surfactant, or combinations of the two in any ratio, optionally together with cationic, amphoteric or zwitterionic surfactants, optionally together
- 20 with soap.

The detergent compositions of the invention also contain one or more detergency builders. The total amount of detergency builder in the compositions will suitably range from 5 to

25 80 wt%, preferably from 10 to 60 wt%.

Suitable inorganic non-phosphorus containing builders include crystalline and amorphous aluminosilicates, for example zeolites as disclosed in GB 1 473 201 (Henkel);

30 amorphous aluminosilicates as disclosed in GB 1 473 202

(Henkel); and mixed crystalline/amorphous aluminosilicates as disclosed in GB 1 470 250 (Henkel); and layered silicates as disclosed in EP 164 514B. Inorganic phosphate builders, for example, sodium orthophosphate, pyrophosphate and
5 tripolyphosphate, may also be present.

Aluminosilicates, include the zeolite used in most commercial particulate detergent compositions, namely zeolite A. Advantageously, however, maximum aluminium
10 zeolite P (zeolite MAP) described and claimed in EP 384 070B (Unilever) may be used. Zeolite MAP is an alkali metal aluminosilicate of the P type having a silicon to aluminium ratio not exceeding 1.33, preferably not exceeding 1.15, and more preferably not exceeding 1.07.

15 Suitably the builder system comprises a zeolite (for example zeolite A) and optionally an alkali metal citrate and/or a crystalline layered silicate (for example SKS-6 ex Hoechst).

20 Examples of phosphorus-containing inorganic detergency builders include the water-soluble salts, especially the alkali metal salts of pyrophosphates, orthophosphates, polyphosphates and phosphonates.

25 The phosphorus containing inorganic builder is preferably pyrophosphate or polyphosphate. Specific examples of inorganic phosphate builders include sodium and potassium tripolyphosphates, orthophosphates and hexametaphosphates.

30 Organic builders that may be present include polycarboxylate polymers such as polyacrylates and acrylic/maleic

copolymers; monomeric polycarboxylates such as citrates, glucomates, oxydisuccinates, glycerol mono-, di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, alkyl- and alkenylmalonates and succinates; and sulphonated fatty acid salts. These materials are preferably present in alkali metal salt, especially sodium salt, form. This list is not intended to be exhaustive.

10

Especially preferred organic builders are citrates, suitably used in amounts of from 5 to 30 wt%, preferably from 10 to 25 wt%; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, preferably from 1 to 10 wt%. Builders, both inorganic and organic, are preferably present in alkali metal salt, especially sodium salt, form.

Detergent compositions according to the invention may also suitably contain a bleach system. Preferably this will include a peroxy bleach compound, for example, an inorganic persalt or an organic peroxyacid, capable of yielding hydrogen peroxide in aqueous solution. Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate. The sodium percarbonate may have a protective coating against destabilisation by moisture. The peroxy bleach compound is suitably present in an amount of from 5 to 35 wt%, preferably from 10 to 25 wt%.

The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. The bleach precursor is

suitably present in an amount of from 1 to 8 wt%, preferably from 2 to 5 wt%. Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and peroxybenzoic acid precursors; and
5 peroxycarbonic acid precursors. An especially preferred bleach precursor suitable for use in the present invention is N,N,N',N'-tetracetyl ethylenediamine (TAED).

A bleach stabiliser (heavy metal sequestrant) may also be
10 present. Suitable bleach stabilisers include ethylenediamine tetraacetate (EDTA), diethylenetriamine pentaacetate (DTPA), ethylenediamine disuccinate (EDDS), and the polyphosphonates such as the Dequests (Trade Mark), ethylenediamine tetramethylene phosphonate (EDTMP) and
15 diethylenetriamine pentamethylene phosphate (DETPMP).

Bleach ingredients are postdosed.

The compositions of the invention may contain alkali metal,
20 preferably sodium, carbonate, in order to increase detergency and ease processing. Sodium carbonate may suitably be present in amounts ranging from 1 to 60 wt%, preferably from 2 to 40 wt%. Sodium carbonate may be included in either or both base granule, and/or may be
25 postdosed.

As previously indicated, sodium silicate may also be present. The amount of sodium silicate may suitably range from 0.1 to 5 wt%. Sodium silicate may be postdosed, for
30 example, as granular sodium disilicate, or as sodium

carbonate/sodium silicate cogranules, for example, Nabion (Trade Mark) 15 ex Rhodia Chimie.

Powder flow may be improved by the incorporation of a small amount of a powder structurant. Examples of powder structurants, some of which may play other roles in the formulation as previously indicated, include, for example, fatty acids (or fatty acid soaps), sugars, acrylate or acrylate/maleate polymers, sodium silicate, and dicarboxylic acids (for example, Sokalan (Trade Mark) DCS ex BASF). One preferred powder structurant is fatty acid soap, suitably present in an amount of from 1 to 5 wt%.

Other materials that may be present in detergent compositions of the invention include antiredeposition agents such as cellulosic polymers; soil release agents; anti-dye-transfer agents; fluorescers; inorganic salts such as sodium sulphate; enzymes (proteases, lipases, amylases, cellulases); dyes; coloured speckles; perfumes; and fabric conditioning compounds. This list is not intended to be exhaustive.

EXAMPLES

An effervescent granule was made with the following composition:

5

Ingredient	Wt%
Citric acid	38
Sodium bicarbonate	42
Sodium carbonate	10
Sodium C ₁₂₋₁₄ alkyl sulphate	5
Polypropyleneglycol	4.5
Silica Gel	0.5
Total	100

The sodium carbonate had a d₅₀ particle size of about 150 microns. The sodium bicarbonate had a d₅₀ particle size of about 120 microns. The citric acid had a d₅₀ particle size of about 250 microns. The sodium C₁₂-C₁₄ alkyl sulphate was in particulate form (with a melting point in excess of 60°C) and had a d₅₀ particle size of about 200 microns.

15 The sodium carbonate, sodium bicarbonate, silica gel, sodium lauryl sulphate and citric acid were mixed together to provide a dry-mix. Polypropylene glycol was then added to produce a wetted mixture. The resultant mixture was then granulated in a sieve-type granulator with 2 mm holes. The extrudates were then sliced into cylinders with a length of approximately 2 mm.

1 parts of the effervescent granule was mixed with 99 parts of a phosphate detergent powder with a formulation given below:

Ingredient	Wt%
Base Powder	
Sodium C ₁₂ alkyl benzene sulphonate	18.0
Sodium tripolyphosphate	12.0
Sodium silicate	7.5
Sodium sulphate	26.9
SCMC	0.50
Optical brightener	0.18
Photobleach	0.03
Silicone	0.02
Salts + moisture	0.5
Post Dosed	
Sodium carbonate	15.0
Sodium sulphate	10.0
Protease	0.24
Amylase	0.10
Sodium perborate monohydrate	1.50
TAED	0.50
Perfume	0.24
Moisture	6.8
Total	100.00

5

The resultant detergent powder had an equilibrium RH at 25°C of 26%, and after 2 weeks storage gave an attractive effervescent action when added to wash water despite the very low level of effervescent granule.

CLAIMS

1. A particulate laundry detergent composition having an equilibrium relative humidity value at 25°C and 1 atmosphere of at least 10% and comprising an effervescent granule, the effervescent granule comprising an acid source, a carbonate source and a desiccant.
2. A detergent composition as claimed in claim 1, which has an equilibrium relative humidity at 25°C and 1 atmosphere of at least 15%.
3. An detergent composition as claimed in claim 1 or claim 2, which has an equilibrium relative humidity at 25°C and 1 atmosphere of at least 20%.
4. A detergent composition as claimed in any preceding claim, wherein the desiccant in the effervescent granule is silica gel.
5. A detergent composition as claimed in any preceding claim, wherein the desiccant is present at a level of from 0.1 to 10 wt%, preferably from 0.2 to 5 wt%, more preferably from 0.3 to 2 wt%, of the granule.
6. A detergent composition as claimed in any preceding claim, wherein the effervescent granule comprises from 20 to 80 wt%, preferably from 30 to 60 wt%, more preferably from 40 to 50 wt% of the carbonate source.

7. A detergent composition as claimed in any preceding claim, wherein the effervescent granule comprises from 10 to 60 wt%, preferably from 20 to 50 wt%, more preferably from 30 to 40 wt% of the acid source.
- 5 8. A detergent composition as claimed in any preceding claim, wherein the carbonate and the acid source make up from 50 to 100 wt%, preferably from 60 to 99 wt%, more preferably from 70 to 95 wt%, most preferably from 10 80 to 90 wt% of the effervescent granule.

